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DEVELOPMENT OF FLEXIBLE POLYMERS
AS THERMAL INSULATION IN SOLID-PROPELLANT
ROCKET MOTORS

Quarterly Progress Report
Contract DA-36-034-ORD-3325 RD
July 1 to September 30, 1962

Submitted to:
Rock Island Arsenal
Rock Island, Illinois

October 1962

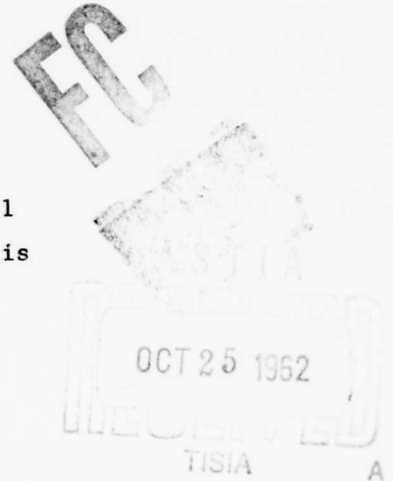
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ABSTRACT

The preparation of flexible furan resins by the polymerization of furfural, furfuryl alcohol, and furan prepolymers by long chain acids (to serve as both catalyst and plasticizer) does not look promising. However, *flexible materials were obtained by* ~~the preparation of co-polymers with the furan prepolymers and epoxy resins or diisocyanates, resulted in flexible materials.~~ *copolymerization of*

Mechanical properties were determined for ~~two of the better insulation materials developed under this program, the 40 per cent castor oil-modified Guardian filled with 40 per cent of asbestos fibers and the 1:1 phenol-formaldehyde:Syl Kem 90 resin, filled with 45 per cent of asbestos fibers.~~ The ultimate elongations were 6 and 0.4 per cent, respectively. However, the mechanical properties of the castor oil-modified Guardian were determined on defective specimens and the elongation of 6 per cent represents only a minimum value.

Four static-motor firings were made, ~~during this report period:~~ Several new formulations were tested. A char rate of 1.7 mil/sec was obtained for an Epon 828:tetrahydrophthalic anhydride:castor oil formulation filled with 40 per cent of asbestos fiber.

Specimens containing other fillers besides pure asbestos fibers were evaluated in motor tests. The 40 per cent castor oil-modified Guardian filled with 20 per cent of asbestos fibers and 20 per cent of potassium oxalate had a char rate of 2.4 mil/sec and showed a slight improvement over the material filled only with 40 per cent of asbestos fiber. The castor oil-modified Guardian filled with 40 per cent of silica-microballoons had a char rate of 5.0 mil/sec, but a very low density, 0.71 gm/cc. The densities of the 40 per cent asbestos and oxalate filled materials are about 1.45 gm/cc. The effect on the char rate of various amounts of asbestos fibers and asbestos fibers - potassium oxalate ratios in the phenol-formaldehyde ~~Syl Kem 90~~ formulation was found to be small.

I. INTRODUCTION

The purpose of thermal insulation is to prevent excessive heating of the structural parts of a missile by the burning propellant. The insulation must be sufficiently flexible to conform to the shape of the structural parts which strain elastically under high operational pressure. If the insulation does not elongate adequately, it fractures, and the hot propellant gases penetrate to the motor case to cause overheating and failure. When the insulation is bonded to the grain or to the motor case, flexibility is essential to prevent fracture of the bond and possible motor failure by side burning.

The purpose of this project is to develop flexible polymers, which, when combined with appropriate fillers, will be suitable for use as thermal insulation in solid propellant rocket motors. Polymeric systems selected for investigation are epoxies, phenolics, melamines, furans, polyurethanes, and polyesters. The investigation includes the modification of commercially available resins, the synthesis of new polymers, and the correlation of polymer structure with performance.

II. RESULTS AND DISCUSSION

A. EPOXY RESINS

1. Mechanical Testing

The exact amount of flexibility needed in the insulation has not been well-defined. Calculations show that the maximum strain of a steel motor case is 0.4 to 0.8 per cent during pressurization and occurs over a period of 0.1 second (time for motor to reach full pressure). Thus, a material having an elongation of 1 per cent under a strain rate of 5 in/in/min should be satisfactory. Greater elongation than 1 per cent is obviously desirable. It is probably safe to predict that a material with an elongation of 100 per cent would be usable, 5-10 per cent may be marginal, while less than 0.8 per cent could not be recommended.

Generally our resins are roughly screened by manual flexing. For those resins found to have superior insulating ability, mechanical properties are determined. One of the best insulations developed to date has been the 40 per cent castor oil-modified Guardian filled with 40 per cent of asbestos fibers. The pure resin is very flexible, but of course, when it is filled with asbestos fibers, its elongation is greatly reduced. Attempts to prepare thin (1/8-inch-thick), asbestos-filled specimens for testing have not been very successful. The average mechanical properties of 3 specimens, visually observed to contain defects on their surfaces, were as follows:

Ultimate Elongation	6 per cent
Ultimate Stress	1500 psi
Young's Modulus	66,500 psi

The fact that the failures were observed to occur at these defects indicates that these values represent only a minimum elongation and tensile strength for this 40 per cent castor oil-modified Guardian filled with 40 per cent asbestos fibers. Attempts to prepare better specimen will be made.

2. Static Motor Firing Tests

Evaluation of flexible epoxy resins in a 5-inch static motor has continued. Several new asbestos-filled formulations were tested as well as old formulations with new filler combinations. A direct comparison of the more promising materials was obtained by retesting them in the same motor.

In motor firing B-72 (Table I), four asbestos-filled, flexible, epoxy resins were tested, along with two of the better commercially available insulations, General Tire and Rubber Company's Gen-Gard V-44 and U. S. Rubber Company's 3015. Because the nitrogen quencher failed to operate at the end of this firing, these six materials were retested in motor firing M-305 (Table II). The nitrogen quench is used to immediately lower the temperature of the motor and to prevent burning of the specimen after the firing is completed. The omission of the quench can result in unreliable char rates for the specimens. The severe alteration in the uncharred materials for specimen V-44, 3015, and XXXXII in motor firing B-72 can be seen in Figure 1. Figure 2 shows the tested specimens from firing M-305.

Gen-Gard V-44 appears to be one of the materials that shows a lower char rate* if the quenching technique is not used. This behavior is related to swelling of the uncharred material. Based on data from previous B-type firings (6500°F flame temperature), the char rate for the V-44 should be somewhat higher, while the char rate for the 3015 should be somewhat lower, than the observed values in firing B-72. Thus, since the reliability of the data in this motor firing are in question, only the char rates in motor firing M-305 are compared.

Because a 5600°F propellant was used in motor firing M-305, the char rates are all lower than the values reported in B-72. The performance of IX-A (Oxiron 200-Empol 1014) is somewhat disappointing in that this particular formulation was one of the best materials in the oxycetylene torch screening test.** Based on previous motor firings, the char rates of the V-44 and 3015 in this firing are perhaps somewhat lower than what might be expected. Nevertheless, the char rate of 1.7 mil/sec for formulation XXXXII (Epon 828:tetrahydrophthalic anhydride:castor oil) was the best of this series. Although a char rate of 1.7 mil/sec may seem abnormally low, visual examination of the uncharred material under the charred layer showed it to be

*The amount of char is determined by subtracting the thickness of the uncharred layer from the initial thickness of the specimen. If this uncharred layer swells, the measured amount of charring will be lower than actual.

**Second Annual Summary Report for this contract, July 31, 1962. Pages 16, 18.

TABLE I
Convergent-Section Motor Firing (B-72)
Results for Filled Epoxy Resins

Code Number	Formulation ^b	Per Cent Asbestos Fiber (3R100)	Density (gm/cc)	Char Rate (mils/sec)	Char Rate × Density
I-A	Epon 828 (1 part) Thiokol LP8 (1 part)	40	1.53	3.7	5.7
II-A	Epon 828 (1 part) Thiokol LP33 (1 part)	40	1.56	3.4	5.3
IX-A	Oxiron 2000 (1 part) Empol 1014 (1 part)	40	1.30	3.6	4.7
XXXXII	Epon 828 (2.8 parts) Tetrahydrophthalic Anhy. (3.0 parts) ^c Castor oil (4.2 parts) ^c	40	1.47	3.4 ^e	5.0
Gen-Gard V-44	General Tire and Rubber Company ^d	--	1.28	3.0 ^e	3.8
3015	U. S. Rubber Company ^d	--	1.24	4.3 ^e	5.3

a. Length of firing: 67.3 sec; flame temperature: 6500°F; medium pressure; motor not quenched at end of firing.

b. Epoxy resin cures were catalyzed with 2.5 per cent benzyldimethyl amine.

c. The THPA and castor oil were heated together in the presence of 0.7 per cent BDMA for 48 hours at 125°C before mixing with Epon 828.

d. Included in firing as a comparative standard.

e. Because of voids and swelling at the center of the specimen, measurements for char rate were made at the edge. (See photograph of tested specimen)

TABLE II
Convergent-Section Motor Firing M-305
Results for Filled Epoxy Resins^a

Code Number	Formulation ^b	Per Cent Asbestos Fiber (3R100)	Density (gm/cc)	Char Rate (mils/sec)	Char Rate X Density
I-A	Epon 828 (1 part) Thiokol LP8 (1 part)	40	1.53	3.4	5.2
II-A	Epon 828 (1 part) Thiokol LP33 (1 part)	40	1.56	2.7	4.2
IX-A	Oxiron 2000 (1 part) Empol 1014 (1 part)	40	1.30	3.2	4.2
XXXXII	Epon 828 (2.8 parts) Tetrahydrophthalic Anhy. (3.0 parts) ^c Castor oil (4.2 parts) ^c	40	1.47	1.7	2.5
Gen-Gard V-44	General Tire and Rubber Company ^d	--	1.28	2.0	2.6
3015	U. S. Rubber Company ^d	--	1.24	2.5	3.1

a. Length of firing: 59.6 sec; flame temperature: 5600°F; high pressure; motor quenched with nitrogen at end of firing.

b. Epoxy resin cures were catalyzed with 2.5 per cent benzyldimethyl amine.

c. The THPA and castor oil were heated together in the presence of 0.7 per cent BDMA for 48 hours at 125°C before mixing with Epon 828.

d. Included in firing as a comparative standard.

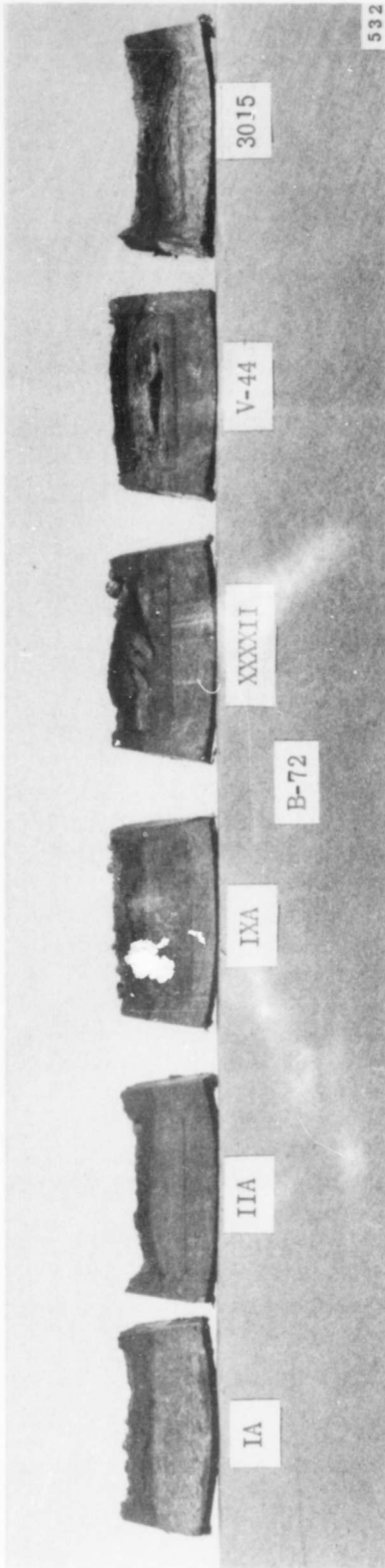


FIGURE 1, B-72

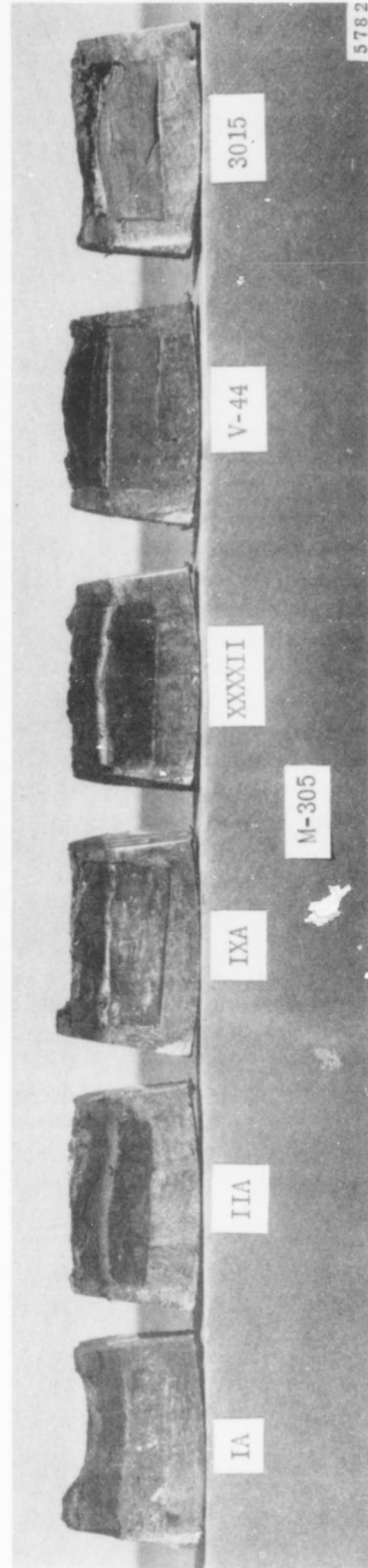


FIGURE 2, M-305
Photographs of Tested Specimens from Motor Firings B-72 and M-305

unchanged with no evidence of swelling or voids. Furthermore, as previously discussed,* this formulation was expected to yield a material of superior performance because (1) the rigid Epon 828:tetrahydrophthalic anhydride formulation has an average char rate of 2.2 mil/sec based on several motor firings and (2) the addition of castor oil to epoxy resins not only increases the flexibility of the resin but also increases the insulation performance (lower char rate). This lower char rate will, however, need verification in additional motor tests. On the basis of this motor firing, the char rate \times density value of 2.5 shows this insulation to be one of the best developed to date.

In firing M-303 (Table III), the effect of several filler combinations was determined. Replacing half the asbestos in the Guardian formulation with potassium oxalate (formulation number XXI C) gave only a marginal improvement in char rate (from 3.2 to 2.9 mil/sec). Replacing half of the asbestos in the 40 per cent castor oil-modified Guardian with potassium oxalate also gave only a marginal improvement (2.6 mil/sec for formulation XXXV versus 2.4 mil/sec for formulation XXII A). When the 40 per cent of asbestos fiber was replaced by silica microballoons (Eccospheres SI) in the 40 per cent castor oil-modified Guardian, the char rate increased from 2.6 (formulation XXXV) to 5.0 mil/sec (formulation XXXXVI). However, the density is significantly reduced from 1.45 gm/cc for the asbestos-filled material to 0.71 gm/cc for the Eccospheres-filled material. Therefore, on the basis of the char rate and density, this material (XXXXVI) is one of the best insulations in this group with a char rate-density factor of 3.6.

The only better material was the 40 per cent castor oil-modified Guardian filled with 20 per cent of asbestos fiber and 20 per cent of potassium oxalate with a char rate-density factor of 3.5. The specimens from motor firing M-303 are shown in Figure 3. It can be observed that the V-44 control which represents one of the best commercially available insulations delaminated with obvious swelling of the uncharred material. This swelling indicates that the actual char rate is probably greater than the 2.8 mil/sec value reported and that, at least in this motor firing, its performance is not as good as indicated.

Based on the results of these motor firings, specimens filled with asbestos-silica microballoons and perhaps even asbestos-potassium

* Second Annual Summary Report for this contract, July 31, 1962. Page 16 and 18.

TABLE III
Convergent-Section Motor Firing M-303
Results for Filled Epoxy Resins^a

Code Number	Formulation ^b	Filler	Density (gm/cc)	Char Rate (mils/sec)	Char Rate X Density
XXI-C	Epon 828 (10 parts) Nadic methyl anhydride (9 parts)	20% asb. fiber	1.55	2.9	4.5
		20% pot. oxalate			
Guardian	Epon 828 (10 parts) Nadic methyl anhydride (9 parts)	40% asb. fiber	1.54	3.2	4.9
		20% asb. fiber			
XXII-A	40% castor oil modified Guardian	20% pot. oxalate	1.46	2.4	3.5
		40% Eccospheres SI			
XXXVI	40% castor oil modified Guardian	40% Eccospheres SI	0.71	5.0	3.6
XXXV	40% castor oil modified Guardian	40% asb. fiber	1.46	2.6	3.8
3016	U. S. Rubber Company ^c	asbestos filler	1.39	2.8 ^d	3.9

a. Length of firing: 61.6 sec; flame temperature: 5600°F; high pressure; motor quenched with nitrogen at end of firing.

b. Epoxy resin cures were catalyzed with 2.5 per cent benzyldimethyl amine.

c. Included in firing as a comparative standard.

d. Because of voids and swelling at the center of the specimen, measurements for char rate were made at the edge. (See photograph of tested specimens)

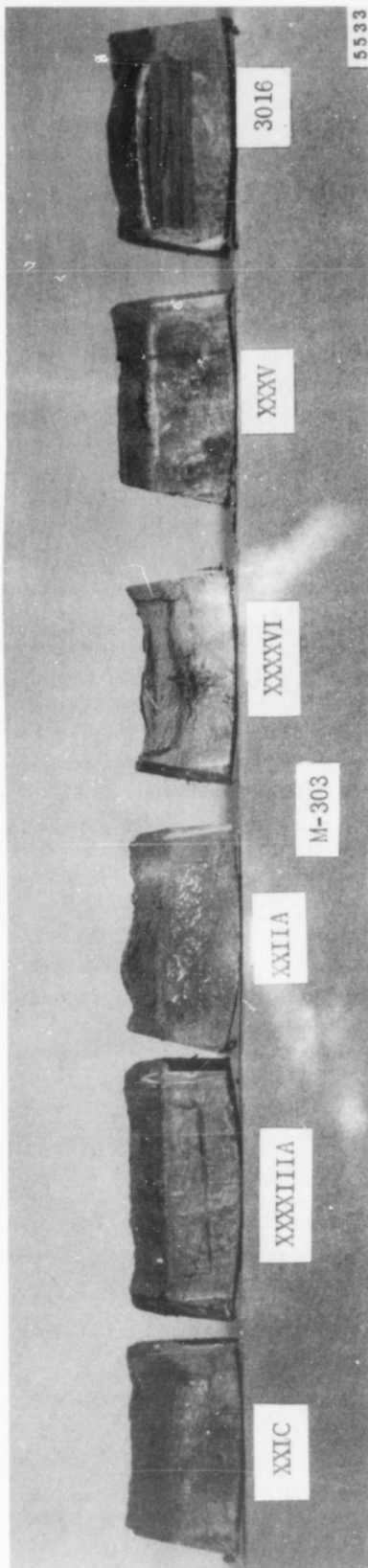


FIGURE 3, M-303

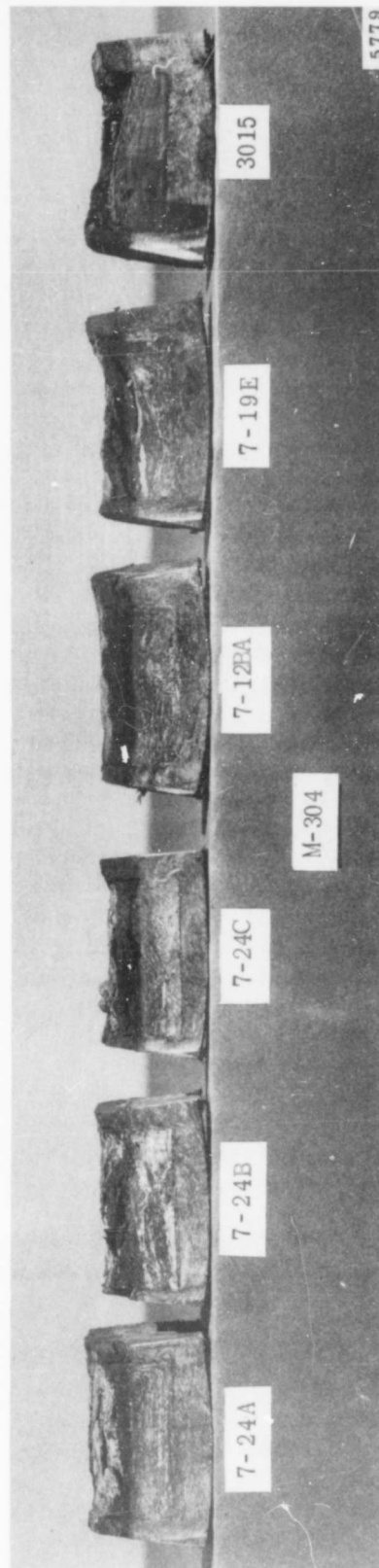


FIGURE 4, M-304

Photographs of Tested Specimens from Motor Firing M-303 and M-304

oxalate-silica microballoons will be studied.

B. PHENOLIC RESINS

1. Mechanical Testing

Since the modified phenol-formaldehyde resin having the best insulation performance (lowest char rate) was the 1:1 phenol-formaldehyde "stage A" resin:Syl Kem 90, the mechanical properties of this material containing 45 per cent of asbestos fibers (3R100) were determined.

The average for three samples is:

Ultimate Elongation	0.36% (17%)
Ultimate Stress	1350 psi (3290 psi)
Young's Modulus	66,500 psi (---)

For comparison, the values for the unfilled material are shown in the parentheses.

Since the elongation of the asbestos filled resin is low, it will be necessary to use a filler which does not reduce the elongation so drastically or use a formulation (for example the 40% phenol-formaldehyde: 60% Syl Kem 90) in which the elongation of the unfilled resin is higher (74%).

2. Static-Motor Firing Tests

A series of the 1:1 phenol-formaldehyde:Syl Kem 90, resins filled with various ratios of asbestos fiber to potassium oxalate were prepared and motor tested. The results of this firing (M-304) are reported in Table IV, and a picture of the tested specimens is shown in Figure 4. All specimens in this test were found to have a char rate of 2.8 mil/sec except for specimen 7-24B which had a char rate of 2.5 mil/sec. On the basis of the char rate-density factor, this latter material would be rated the best of the phenolics tested in this series, but it was inferior to U. S. Rubber Company's 3015. It is somewhat surprising that all the specimens performed essentially the same in this motor firing. Some of the materials will be retested to confirm these results.

TABLE IV
Convergent-Section Motor Firing M-304
Results for Filled Phenolic Resins^a

<u>Code Number</u>	<u>Formulation</u>	<u>Per Cent Filler</u>	<u>Density (gm/cc)</u>	<u>Char Rate (mils/sec)</u>	<u>Char Rate × Density</u>
7-24A	Phenol-formaldehyde (1 part) Syl Kem 90 (1 part)	33.8% asb. fiber 11.2% pot. oxalate	1.54	2.8	4.3
7-24B	"	11.2% asb. fiber 33.8% pot. oxalate	1.52	2.5	3.8
7-24C	"	50% asb. fiber	1.60	2.8	4.5
7-12BA	"	45% asb. fiber	1.51	2.8	4.2
7-19E	"	22.5% asb. fiber 22.5% pot. oxalate	1.50	2.8	4.2
3015	U. S. Rubber Company ^b	--	1.24	2.8 ^c	3.5

a. Length of firing: 72.7 sec; flame temperature: 5600°F; medium pressure; motor quenched with nitrogen at end of firing.

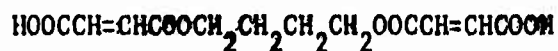
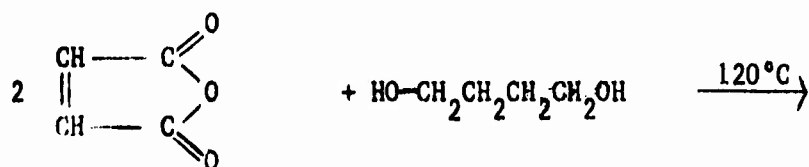
b. Included in firing as a comparative standard.

c. Because of voids and swelling at the center of the specimen, measurements for the char rate were made at the edge. (See photograph of tested specimens.)

C. FURAN RESIN

Several approaches have been followed in our attempts to prepare flexible furan resins. These involve (1) the polymerization of furfural and furfuryl alcohol by long chain acid catalysts which may act as plasticizers, (2) the polymerization of furan prepolymers by these acidic catalysts, and (3) the co-polymerization of furan prepolymers with epoxy resins and polyisocyanates. The results are summarized in Tables V through XI.

The acid catalyst referred to as diacid 150 was prepared by heating one mole of 1,4-butanediol with two moles of maleic anhydride at 120°C for four hours with stirring.



Titration of the product with NaOH gave an experimental equivalent weight of 142; the calculated equivalent weight for this diacid is 143. The infrared spectrum of the product showed a strong ester absorption and no anhydride absorption.

Several commercial furan prepolymers were studied. The exact structures of these prepolymers are not known, but in general, they are prepared by partially polymerizing furfuryl alcohol with an acid catalyst. When the desired viscosity is reached, the reaction is stopped by the addition of base. The prepolymer can be stored for extended periods and further polymerized by adding more acid.

Furfural was not polymerized by diacid 150 or Empol 1014 (a C₃₆ diacid). Furfuryl alcohol could be polymerized by the diacid 150, but only to rigid, brittle resins. Empol 1014 was not effective.

Attempts to polymerize the various furan prepolymers with Empol 1014 resulted in either incompletely cured viscous liquid or foamed, rigid

materials. When diacid 150 was used to cure these prepolymers, generally a hard, rigid product was obtained. However, formulations 73 (Table VIII) and 77, 78, 79 (Table IX) were cured to soft, flexible materials. Whether these materials become rigid on further heating or at ambient temperature is being investigated.

If these furan prepolymers contain two or more free hydroxyl groups per molecule, it may be possible to cure them with epoxy resin or polyisocyanates. The formulations using Atlas' furfuryl alcohol prepolymer (Table X) show some promise and will be investigated further. Both Araldite DP437 and Dow Corning's QZ-8-0914 (epoxy resins) yield very flexible but jelly-like materials.

TABLE V

Polymerization of Furfural with Acid Catalysts

<u>Formulation</u>		<u>Cure Conditions</u>	<u>Results</u>
<u>Furfural/Empol 1014</u>			
1A	9:1	Samples were placed in the oven for 18 hrs at 90°C	Samples were not compatible and did not cure
2A	8:2		
3A	7:3		
4A	6:4		
<u>Furfural/Diacid 150</u>			
5A	9:1	Left at room temperature overnight and then placed in an oven for 18 hrs. at 50°C	Samples did not cure
6A	8:2		
7A	7:3		
8A	6:4		

TABLE VI
Polymerization of Furfuryl Alcohol with Acid Catalysts

<u>Formulation</u>		<u>Cure Conditions</u>	<u>Results</u>
<u>Furfuryl Alcohol/Diacid 150</u>		Samples were cured in an oven at 100°C	All of the samples foamed on curing and were rigid
9A	9:1		
10A	8:2		
11A	7:3		
12A	6:4		
<u>Furfuryl Alcohol/Diacid 150</u>		Samples were left at room temperature overnight and then placed in an oven at 50°C for 18 hours	All of the samples cured to hard and brittle resins
13A	9:1		
14A	8:2		
15A	7:3		
16A	6:4		
<u>Furfuryl Alcohol/Empol 1014</u>		The samples were placed in an oven at 40°C for several hours and then at 100°C for 18 hours	All samples were syrupy and incompletely cured
17A	9:1		
18A	8:2		
19A	7:3		
20A	6:4		

TABLE VII

Polymerization of Furfuryl Alcohol Prepolymer (Atlas)
with Acid Catalysts

<u>Formulation</u>		<u>Cure Conditions</u>	<u>Results</u>
<u>Furan Prepolymer/Embol 1014</u>			
43	9:1	10 hours at 120°C	Very viscous liquid, surface skin
44	8:2	" "	Foamed, cured material rigid and brittle
45	7:3	" "	" "
46	6:4	" "	" "
<u>Furan Prepolymer/Diacid 150</u>			
47	9:1	10 hours at 120°C	Cured, foamed, slightly flexible, brittle
48	8:2	" "	Cured, foamed, rigid, brittle
49	7:3	" "	" " "
50	6:4	" "	" " "

TABLE VIII

Polymerization of Furan Prepolymer
(Durez) with Acid Catalysts

<u>Formulation</u>		<u>Cure Conditions</u>	<u>Results</u>
<u>Furan Prepolymer/Empol 1014</u>			
51	9:1	18 hours at 120°C	Empol 1014 not compatible with furan prepolymer
52	8:2		
53	7:3		
54	6:4		
<u>Furan Prepolymer/Diacid-150</u>			
55	9:1	18 hours at 120°C	Rigid, foamed, brittle
56	8:2		" " "
57	7:3		" " "
58	6:4		" " "
<u>Furan Prepolymer/Diacid-150</u>			
73	9:1	18 hours at 50°C then 18 hours at 70°C	Initially viscous. On further curing, became very flexible and soft
74	8:2	18 hours at 50°C	Rigid, foamed, brittle
75	7:3	" "	Rigid, foamed, brittle
76	6:4	" "	Rigid, foamed, brittle

TABLE IX

Polymerization of Furan Prepolymer
(Jet-Kote X-3M) with Acid Catalysts

<u>Formulation</u>		<u>Cure Conditions</u>	<u>Results</u>
<u>Jet-Kote X-3M/Empol 1014</u>			
59	9:1	18 hours at 120°C	Viscous liquid
60	8:2		Viscous liquid
61	7:3		Viscous liquid
62	6:4		Viscous liquid
<u>Jet-Kote X-3M/Diacid-150</u>			
63	9:1	18 hours at 120°C	Cured, flexible, brittle
64	8:2		Cured, slightly flexible, brittle
65	7:3		Cured, rigid, brittle
66	6:4		Cured, rigid, brittle
<u>Jet-Kote X-3M/Diacid-150</u>			
77	9.5:0.5	18 hours at 50°C	Cured, flexible, soft
78	9:1		Cured, flexible, soft
79	8.5:1.5		Cured, flexible, soft

TABLE X

Co-Polymerization of Furfuryl Alcohol Prepolymer (Atlas) with
Epoxy Resins and Polyisocyanates

<u>Formulation</u>		<u>Cure Conditions</u>	<u>Results</u>
<u>Furan Prepolymer/QZ-8-0914 (epoxy)</u>			
1	1:2	8 hours at 100°C	All samples cured to a flexible, jelly-like material
2	1:1		
3	2:1		
<u>Furan Prepolymer/Araldite DP437 (epoxy)</u>			
4	1:2	8 hours at 100°C	All samples cured to a flexible, jelly-like material
5	1:1		
6	2:1		
<u>Furan Prepolymer/Epon 828 (epoxy)</u>			
7	1:2	8 hours at 100°C	Rigid and brittle
8	1:1		" " "
9	2:1		Soft and flexible
<u>Furan Prepolymer/Hexamethylene Diisocyanate</u>			
10	1:2	8 hours at 100°C	Slightly flexible, hard
11	1:1		" " "
12	2:1		Rigid and brittle
<u>Furan Prepolymer/Toluene-2,4-Diisocyanate</u>			
13	1:2	8 hours at 100°C	Foamed, rigid
14	1:1		" "
15	2:1		" "

TABLE XI

Co-Polymerization of Furan Prepolymer (Durez) with
Epoxy Resins and Polyisocyanates

<u>Formulation</u>		<u>Cure Conditions</u>	<u>Results</u>
<u>Furan Prepolymer/QZ-0914 (epoxy)</u>			
16	1:2	24 hours at 100°C	Flexible but soft
17	1:1		Very viscous liquid
18	2:1		" " "
<u>Furan Prepolymer/Araldite DP437 (epoxy)</u>			
19	1:2	24 hours at 100°C	Very viscous liquid
20	1:1		" " "
21	2:1		" " "
<u>Furan Prepolymer/Epon 828 (epoxy)</u>			
22	1:2	24 hours at 100°C	Rigid and brittle
23	1:1		" " "
24	2:1		" " "
<u>Furan Prepolymer/Hexamethylene Diisocyanates</u>			
25	1:2	24 hours at 100°C	Slightly flexible
26	1:1		Flexible but brittle
27	2:1		Rigid, brittle
<u>Furan Prepolymer/Toluene-2,4-Diisocyanate</u>			
28	1:2	24 hours at 100°C	Foamed, rigid
29	1:1		" "
30	2:1		" "

III. SUMMARY OF INSULATION WORK

A brief summary of the status of this program to date is reported here. Flexible epoxy, phenolic, and urethane resins were developed for insulation studies. Asbestos fiber-filled moldings were prepared and evaluated for insulation performance in the oxyacetylene torch and in static-motor firings. In addition to asbestos fibers, potassium oxalate, asbestos fiber-potassium oxalate, silica micro-balloons, magnesium carbonate, and asbestos powder have been investigated as possible fillers for the more promising resins.

Considerable effort was devoted to the preparation of flexible melamine resins. Although a few flexible formulations were developed, asbestos-filled moldings have not been successfully prepared. Studies with the furan resins have yielded several flexible materials by co-polymerizing furan resin prepolymers with epoxy resins and diisocyanates.

The best insulations developed to date in this program are summarized in Table XII. The better commercially available motor-case insulations have char rates of approximately 3.0 mil/sec in our test motors. In addition to the insulation performance, the density of the material is also an important consideration in the selection of insulation for missile applications. Other properties being equivalent, the product of char rate and density allows a numerical rating of the material according to over-all effectiveness, with the lowest number representing the best material. The volume requirements of the insulation are neglected.*

It should be emphasized that (1) the data in Table XII represent only a limited number of tests; (2) char rate differences of 0.5 mil/sec are probably not significant when based on one or two tests; and (3) differences in the propellant used, time duration of the motor firing, pressure, and specimen position, make it difficult to compare materials tested in different motors. U. S. Rubber Company's 3015 and 3016, and General Tire and Rubber Company's Gen-Gard V-44 were used as comparative standards and represent some of the best commercially available insulation materials.

* This point is discussed in the Second Annual Summary Report, This Contract, July 31, 1962, pp. 40, 73.

TABLE XII
Summary of Best Insulation Developed to Date Under This Program

Code Number	Formulation	Filler Content	Density (gm/cc)	Motor Firing Conditions		Char Rate (mils/sec)	Char Rate x Density
				Position	Flame Temp (°F)		
XXXXII	Epon 828/THPA ^a /castor oil (2.8/3.0/4.2)	40% asbestos fibers ^b	1.47	Convergent	5600	1.7(2.5) ^c (2.0) ^d	2.5(3.1) ^c (2.6) ^d
XXII-A	40% castor oil modified Guardian, varying NMA ^e	20% asbestos fiber ^b 20% pot. oxalate	1.46	Convergent	5600	2.4(2.8) ^f	3.5(3.9) ^f
XXIV	40% castor oil modified Guardian, varying NMA ^e	40% asbestos fiber ^b	1.46	Convergent	5600	2.6(2.8) ^f	3.8(3.9) ^f
XIV-F	40% castor oil modified Guardian, varying NMA ^e	41% asbestos fiber ^b	1.41	Convergent	6500	3.0(3.2) ^c	4.2(4.0) ^c
X-B	Araldite DP-437/NMA (1.0/1.0)	40% asbestos fibers ^b	1.52	Convergent	5600	2.4(2.6) ^c	3.6(3.2) ^c
X-A	Araldite DP-437/NMA (1.0/1.0)	40% asbestos fibers ^b	1.52	Peripheral	5600	2.4(2.7) ^c	3.6(3.3) ^c
7-19-E	Phenol-formaldehyde stage "A" resin/Syl-Kem 90 (1.0/1.0)	22.5% asbestos fiber ^b 22.5% pot. oxalate	1.50	Convergent	6500	3.3(3.5) ^c	5.0(4.3) ^c
7-19-E	Phenol-formaldehyde stage "A" resin/Syl-Kem 90 (1.0/1.0)	22.5% asbestos fiber ^b 22.5% pot. oxalate	1.50	Convergent	5600	2.1(3.1) ^c	3.1(3.8) ^c
7-19-E	Phenol-formaldehyde stage "A" resin/Syl-Kem 90 (1.0/1.0)	22.5% asbestos fiber ^b 22.5% pot. oxalate	1.50	Convergent	5600	2.8(2.8) ^c	4.2(3.5) ^c
7-16-B	Nonyl phenol-formaldehyde "A" resin/Syl-Kem 90 (3.0/2.0)	45% asbestos fiber ^b	1.37	Convergent	5600	2.7(2.6) ^c	3.7(3.2) ^c

- a. Tetrahydrophthalic anhydride
b. H. K. Porter Company asbestos fiber, 3R100
c. Values for U. S. Rubber Company's 3015 insulation tested at the same time as a comparative standard
d. Values for General Tire and Rubber Company's Gen-Gard V-44 insulation tested at the same time as a comparative standard.
e. The 40% castor oil modified Guardian formulation is: Epon 828/Nadic methyl anhydride/castor oil (1.00/1.29/1.52)
f. Values for U. S. Rubber Company's 3016 insulation tested at the same time as a comparative standard.

IV. FUTURE WORK

During the next year of this program, the following general areas of work will be emphasized:

(1) Continuation of the development of flexible resins of the furan, melamine, polyester and urethane types;

(2) Study fillers other than asbestos, or combinations of other fillers with asbestos, in the best resins developed to date (epoxy and phenolic);

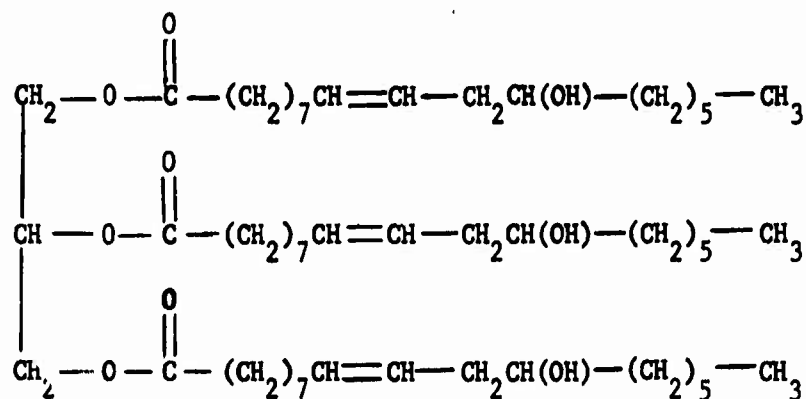
(3) Screen newly developed resins (filled type) by means of the oxyacetylene torch test and test by static motor firings the most promising candidates; and

(4) Furnish the Rock Island Arsenal Laboratory approximately one pound samples of several of the best flexible resins developed under the contract for use in compounding studies.

APPENDIX

Formulary

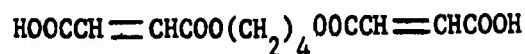
1. Araldite DP-437 (Ciba): An inherently flexible, liquid epoxy resin, epoxy equivalent 285.
2. Asbestos fiber 3R100 (H.K. Porter Co.): Chrysolite asbestos fibers.
3. BDMA (Miles Chemical Company): Benzyldimethyl amine.
4. Castor oil (The Baker Castor Oil Co.):



5. Castor oil-modified Guardian (Atlantic Research Corp.)

40% modification
1.00 part Epon 828
1.29 parts NMA
1.52 parts castor oil

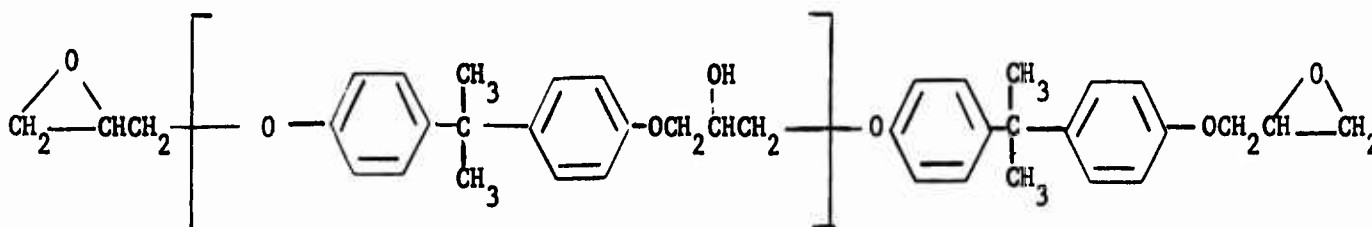
6. Diacid-150



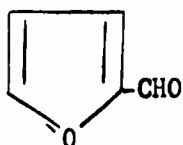
7. Eccospheres SI (Emerson and Cuming): hollow glass microspheres
8. Empol 1014 (Emery): A mixture of 95 per cent of C_{36} diacid, 4 per cent of C_{54} triacid, and 1 per cent of a monobasic acid.

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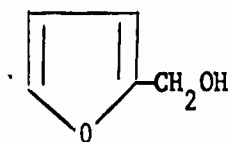
9. Epon 828 (Shell Chemical Corp.): Liquid epoxy resin; epoxy equivalent 180-195.



10. (a) Furan prepolymer (Atlas Mineral Products Co.) - a low viscosity (800-1000 cps) furfuryl alcohol polymer
- (b) Furan prepolymer (Hooker Chemical Corp, Durez) - a low viscosity (200cps) furfuryl alcohol resin (Code No. 16470).
- (c) Furan Prepolymer - "Jet-Kote" X-3M (Furane Plastics Co.) - Nature of the resin unknown.
11. Furfural (Fisher Scientific Company)



12. Furfuryl alcohol (Eastman Organic Chemicals)



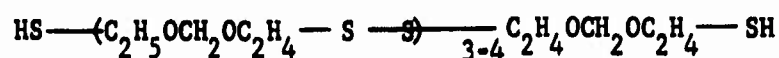
13. Gen-Gard V-44 (The General Tire and Rubber Company): Asbestos-filled butadiene-acrylonitrile rubber insulation.
14. Guardian (Atlantic Research Corporation): This material was referred to previously as Standard Guardian. Its formulation is:

10 parts Epon 828
9 parts NMA

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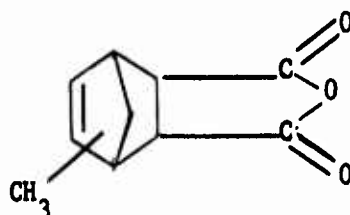
15. Hexamethylene diisocyanate (Mobay Chemical Company): $\text{OCN}(\text{CH}_2)_6\text{NCO}$

16. LP-8 (Thiokol): A liquid polysulfide with the following average structure and which some side mercaptan groups occur occasionally:

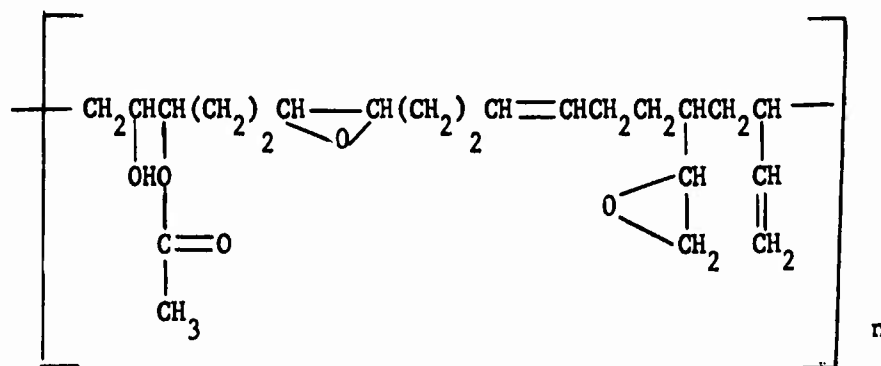


17. LP-33 (Thiokol): Similar to LP8 but the molecule is less cross-linked and the repeating unit occurs 6 times per molecule.

18. Nadic methyl anhydride (National Aniline):



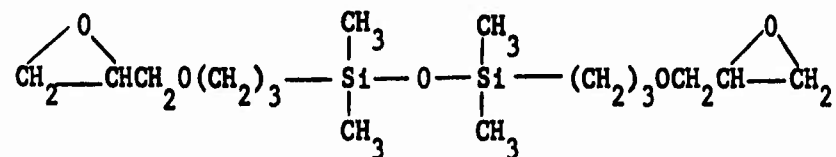
19. Oxiron 2000 (Food Machinery and Chemical Corporation):
Epoxidized polyolefin:



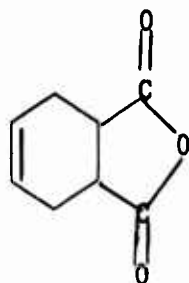
20. Potassium oxalate (Fisher, Certified): $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$

21. QZ-8-0914 (Dow Corning): Technical grade of Syl-Kem 90.
See following structure.

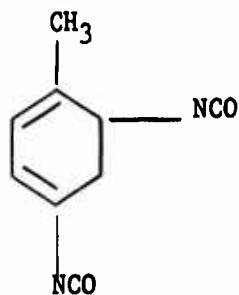
22. Syl-Kem 90 (Dow Corning): A liquid epoxy resin.



23. THPA, tetrahydrophthalic anhydride (Allied Chemical):



24. Toluene diisocyanate (Du Pont, Hylene T):



25. U. S. Rubber Company's 3015 insulation (U.S. Rubber): One of the best commercially available insulations. Used as comparative standard in this work. Potassium oxalate filler.
26. U. S. Rubber Company's 3016 insulation (U.S. Rubber): One of the best commercially available insulations. Used as a comparative standard in this work. Asbestos fiber filler.

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<p>Atlantic Research Corporation, Alexandria, Virginia. DEVELOPMENT OF FLEXIBLE POLYMERS AS THERMAL INSULATION IN SOLID-PROPELLANT ROCKET MOTORS. T. R. Walton and E. B. Simmons. Quarterly Progress Report, October 8, 1962, (July 1 to September 30, 1962). Contract No. DA-36-034-ORD3325-RD, Rock Island Arsenal.</p> <p>Unclassified Report</p> <p>Attempts to prepare flexible furan resins from furfural, furfuryl alcohol, and furan prepolymers are described. Mechanical properties were determined for an asbestos filled epoxy resin and an asbestos filled phenolic resin. Four static-motor firings</p>	<p>UNCLASSIFIED</p> <ol style="list-style-type: none"> 1. Solid propellant motor case insulation. 2. Flexible polymers. 3. Flexible insulation. 4. Epoxy resins. 5. Phenolic resins. 6. Furan resins. 7. Reinforcing materials. 8. Polymer mechanical properties. 9. Static motor testing of thermal insulation. <p>UNCLASSIFIED</p>	<p>Atlantic Research Corporation, Alexandria, Virginia. DEVELOPMENT OF FLEXIBLE POLYMERS AS THERMAL INSULATION IN SOLID-PROPELLANT ROCKET MOTORS. T. R. Walton and E. B. Simmons. Quarterly Progress Report, October 8, 1962, (July 1 to September 30, 1962). Contract No. DA-36-034-ORD3325-RD, Rock Island Arsenal.</p> <p>Unclassified Report</p> <p>Attempts to prepare flexible furan resins from furfural, furfuryl alcohol, and furan prepolymers are described. Mechanical properties were determined for an asbestos filled epoxy resin and an asbestos filled phenolic resin. Four static-motor firings</p>	<p>UNCLASSIFIED</p> <ol style="list-style-type: none"> 1. Solid propellant motor case insulation. 2. Flexible polymers. 3. Flexible insulation. 4. Epoxy resins. 5. Phenolic resins. 6. Furan resins. 7. Reinforcing materials. 8. Polymer mechanical properties. 9. Static motor testing of thermal insulation. <p>UNCLASSIFIED</p>
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